



REFRACTORY METAL/SILICIDE MULTIPHASE SYSTEMS FOR HIGH-TEMPERATURE STRUCTURAL APPLICATIONS

M.G. Mendiratta, S.K. Menon, and T.A. Parthasarathy
Air Force Research Laboratory (*UES),
Materials and Manufacturing Directorate, AFRL/MLLM,
Wright-Patterson AFB, OH 45433-7817

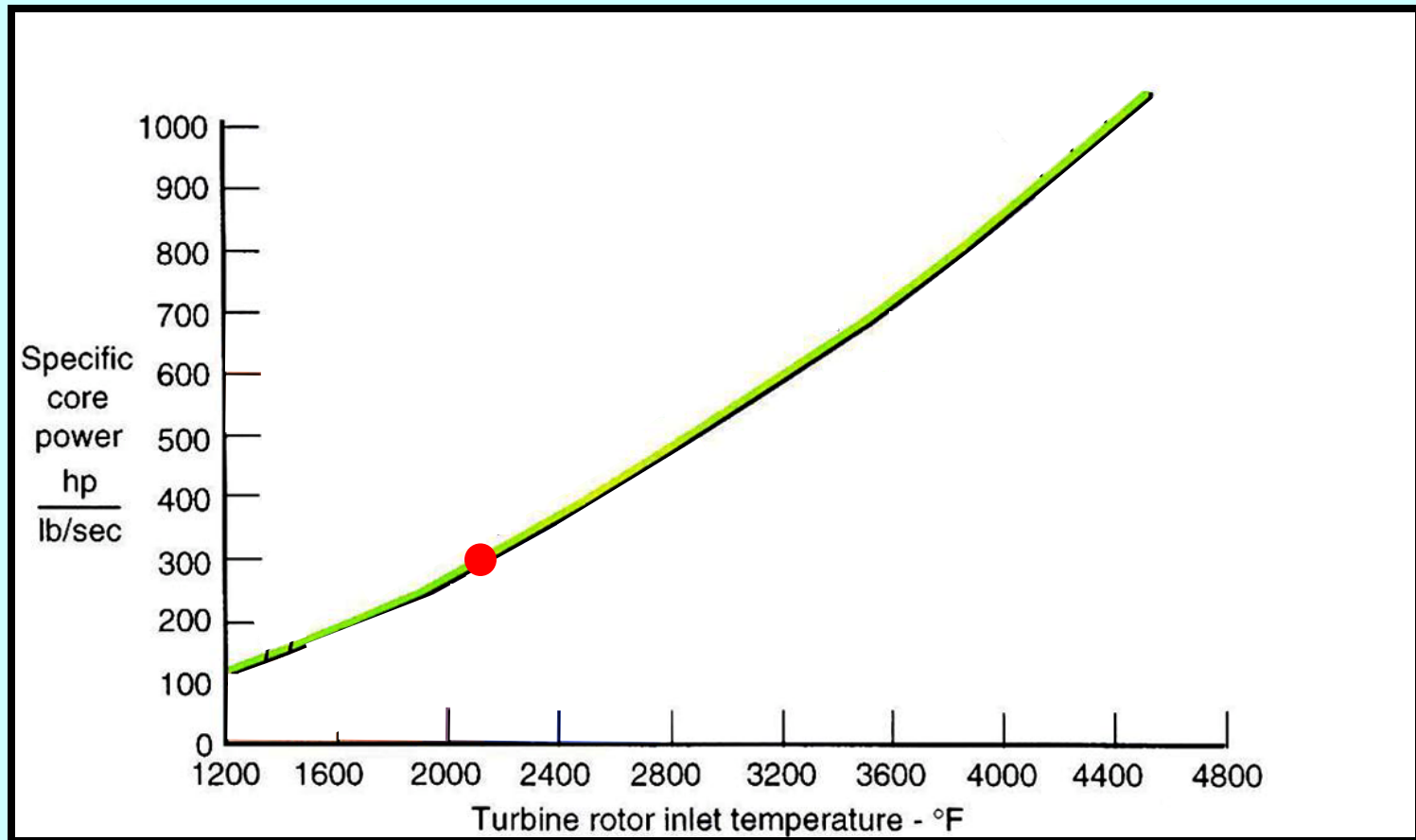
Technical Interactions:

P.L. Martin, D.M. Dimiduk, AFRL/MLLM, Wright-Patterson AFB OH
Mel Jackson and Colleagues, GE CRD, USA
Doug Berczik and Colleagues, P&W, USA

**UES, Inc., Dayton OH 45432 - under USAF Contract #-F33615-01-C-5214*

Report Documentation Page				Form Approved OMB No. 0704-0188	
Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.					
1. REPORT DATE 18 MAR 2004		2. REPORT TYPE N/A		3. DATES COVERED -	
4. TITLE AND SUBTITLE Refractory Metal/Silicide Multiphase Systems For High-Temperature Structural Applications				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Air Force Research Laboratory (*UES), Materials and Manufacturing Directorate, AFRL/MLLM, Wright-Patterson AFB, OH 45433-7817				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES See also ADM001672., The original document contains color images.					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 22	19a. NAME OF RESPONSIBLE PERSON
a. REPORT NATO/unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

OPERATING TEMPERATURE VS SPECIFIC CORE POWER



High Operating Temperature \Rightarrow High Thrust

High Specific Strength \Rightarrow High Structural Efficiency

ADVANCED AIRCRAFT ENGINES – HOT SECTION MATERIALS

Temperature

600°C to 1350°C

Properties

Oxidation Resistance

Toughness

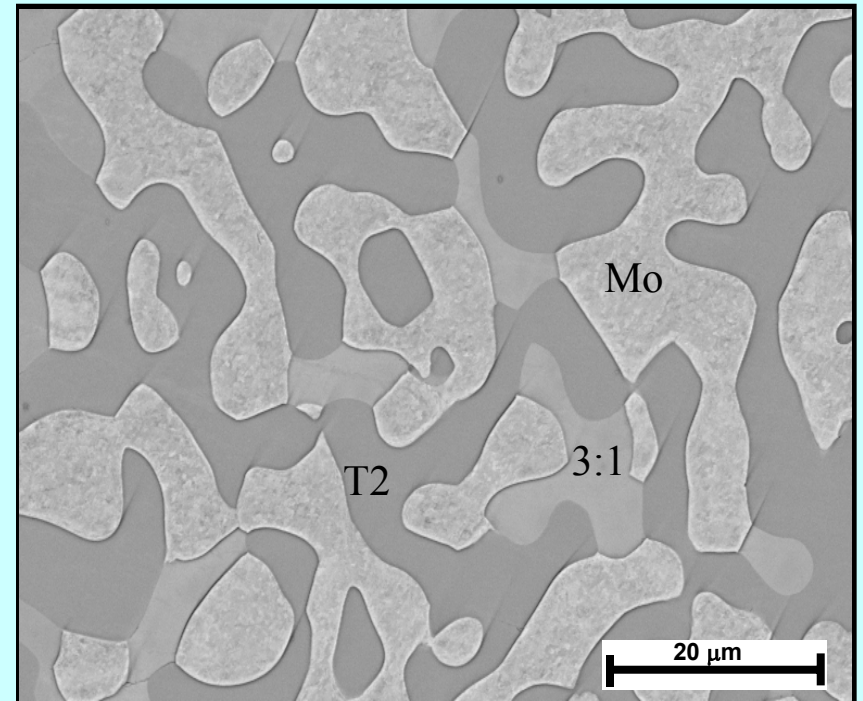
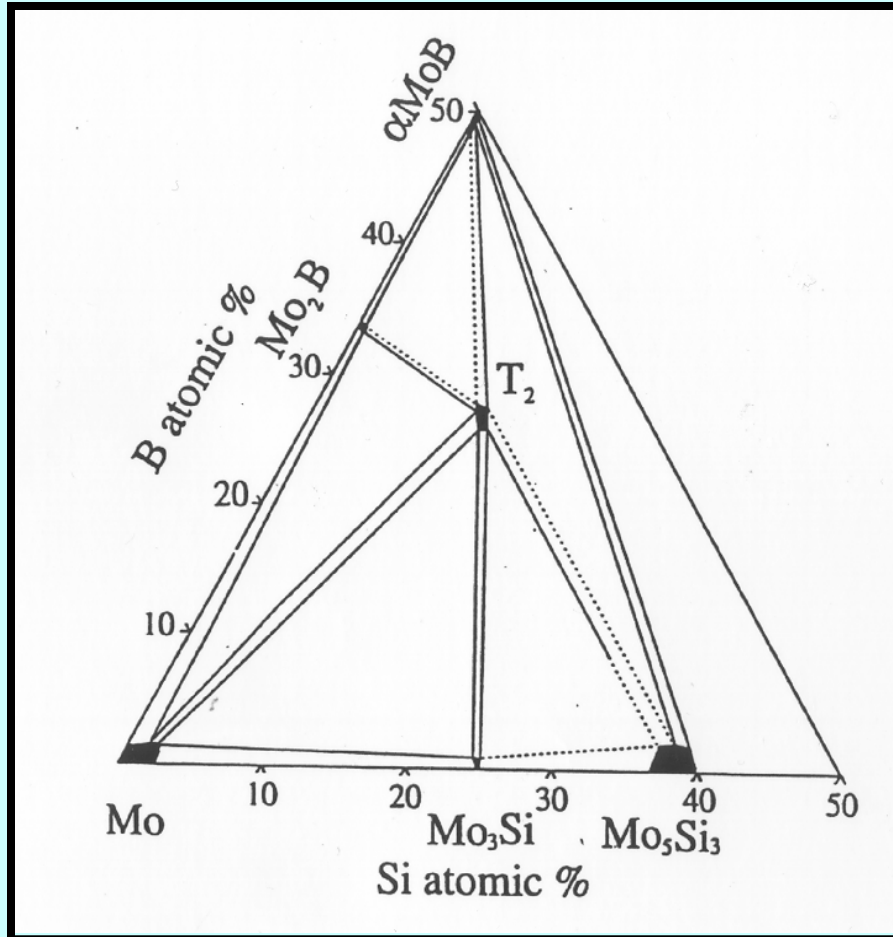
Strength / Creep Resistance

Candidates

Mo- Si - B

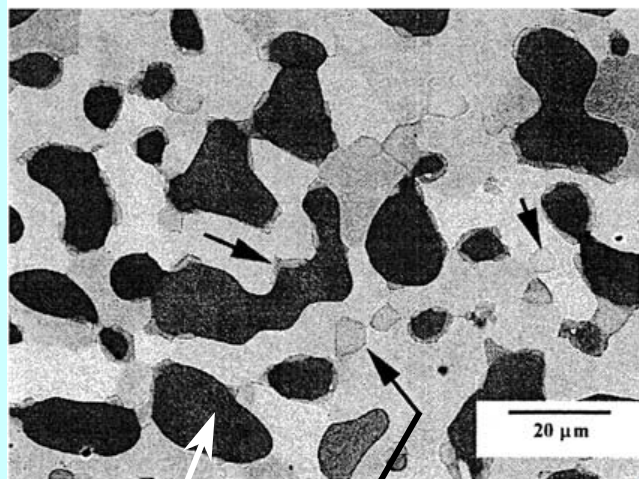
Nb - Ti - Cr - Si - Al -

Mo-Si-B PHASE DIAGRAM (1600°C) & MICROSTRUCTURE



PHASE SPACE OF INTEREST @ 1400°C: Nb/Nb Silicide Alloys

45Nb-26Ti-6.7Cr-12.6Si-1.9Hf-1.9Al-X

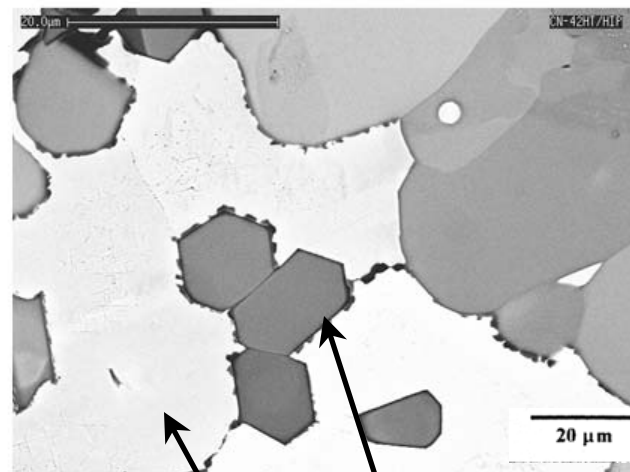


Nb₅Si₃

Ti₅Si₃

20 μm

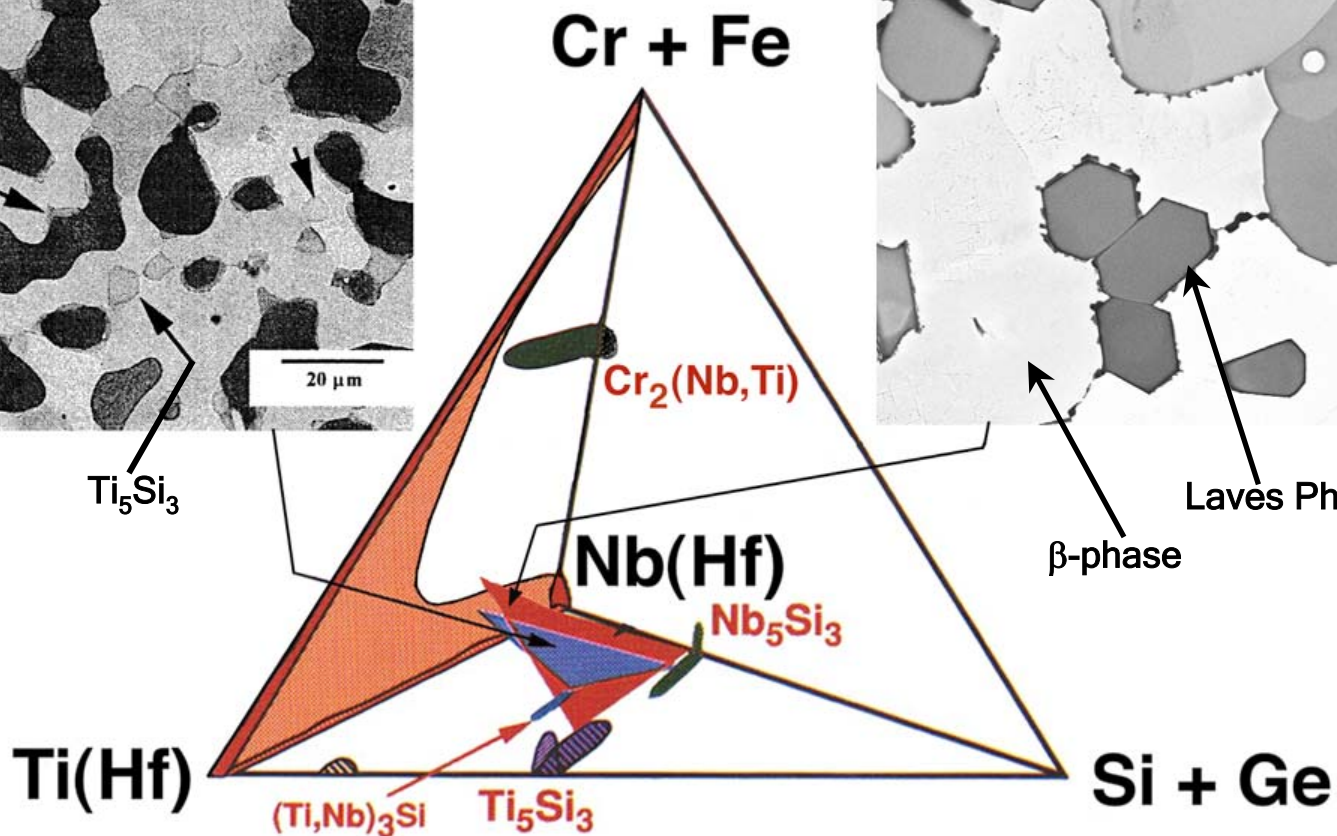
47Nb-21.5Ti-9Cr-17.5Si-2Hf-2Al-X



β-phase

Laves Phase

20 μm

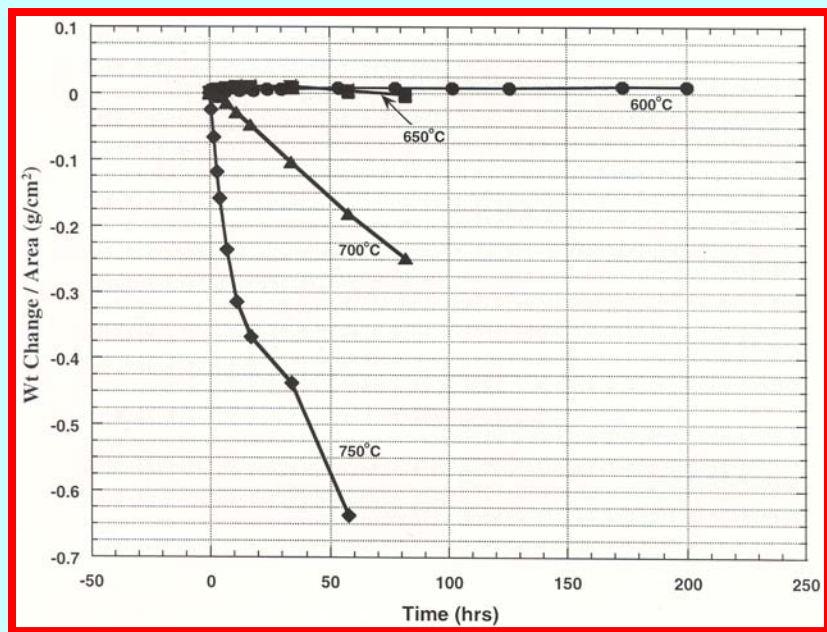


PHYSICAL PROPERTIES*

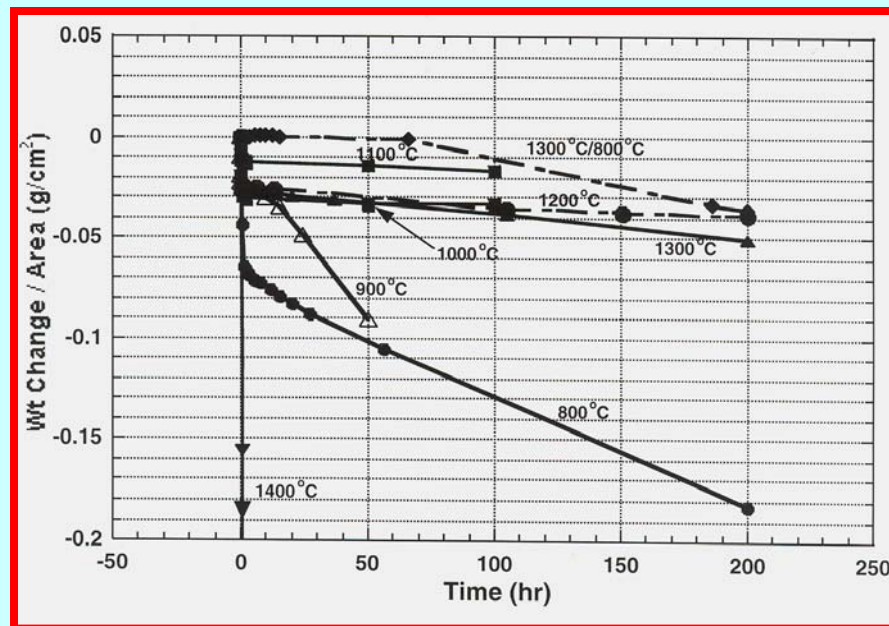
PROPERTY	Mo-Si-B	Nb-Ti-Cr-Hf-Al-Sn
<i>Density</i>	~ 9.5 Mg/m³	~ 7 Mg/m³
<i>Melting Temperature</i>	~ 2100°C	~ 1700 - 1800°C
<i>Thermal Expansion</i>	7 - 11x10⁻⁶ μm/μm	8.1 - 8.6x10⁻⁶ μm/μm
<i>Elastic Modulus</i>	327 - 171 GPa	158-130 GPa
<i>Thermal Conductivity</i>	49.9 - 112 W/m-K	8.6 - 28 W/m-K
<i>Thermal Fatigue Resistance = $k K_{IC} / E\alpha$</i>	~ 15 x 10⁴ (RT)	~ 7 x 10⁴ (RT)
<i>Impact Resistance</i>	~ Very Low	Low (Acceptable)

***Sources: Literature, GE CRD, P&W (Private Communications), NASA**

CYCLIC OXIDATION KINETICS OF Mo-12Si-12B FROM 600-1400°C

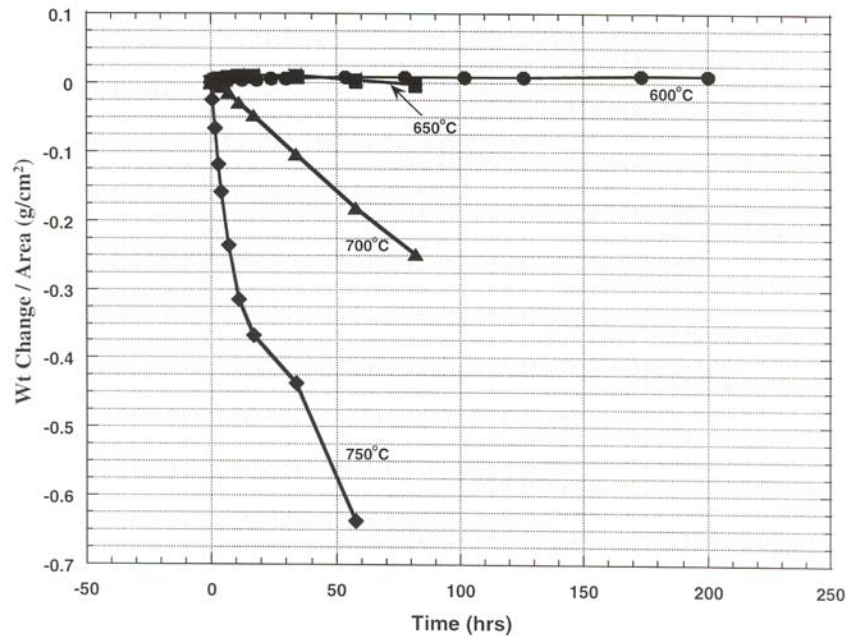


600 - 700°C

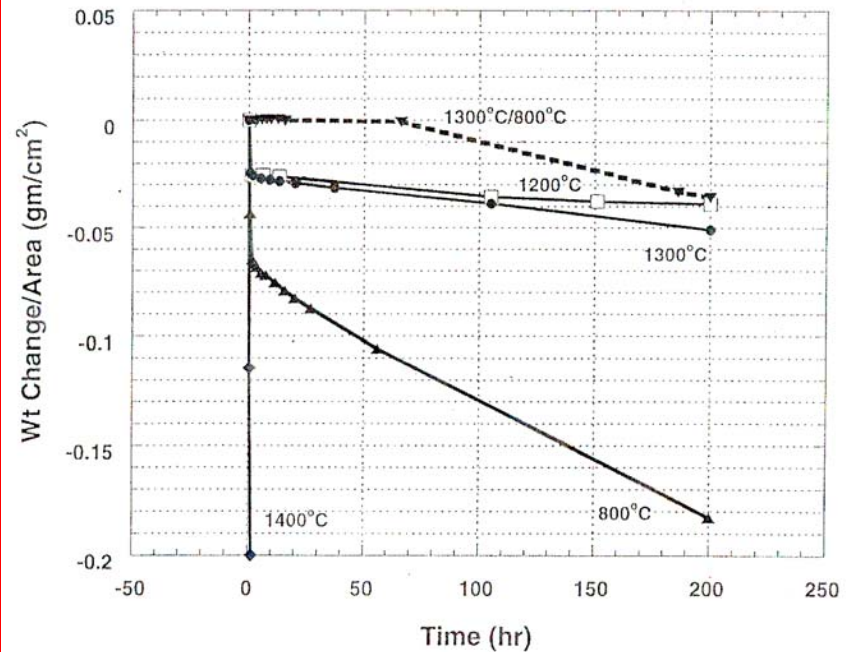


800 - 1400°C

CYCLIC OXIDATION KINETICS OF Mo-12Si-12B FROM 600-1400°C

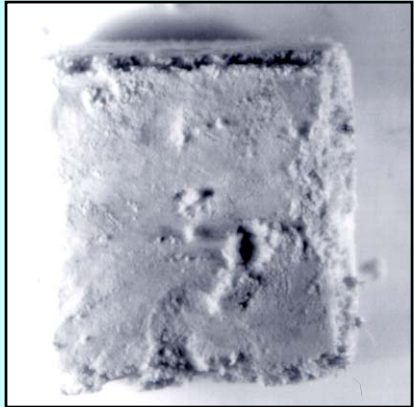


600 - 700°C

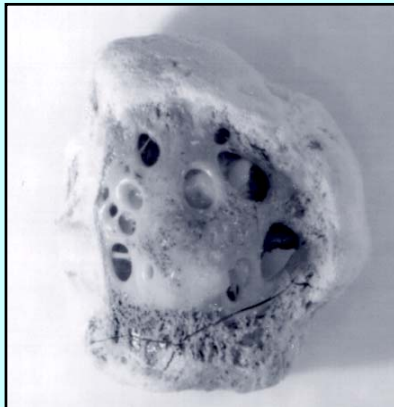


800 - 1400°C

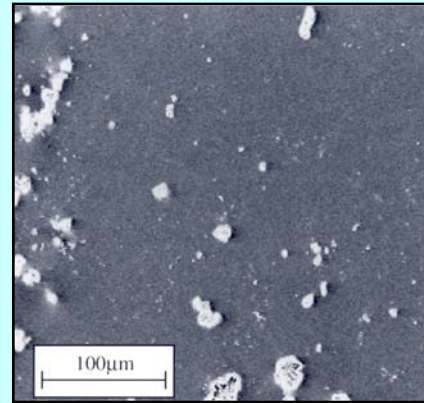
OXIDE SCALE MICROSTRUCTURES: Mo-12Si-12B



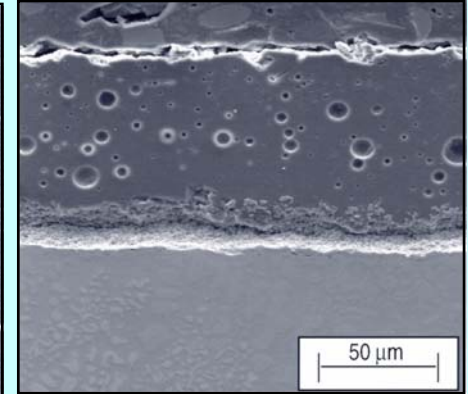
700°C / 82 h



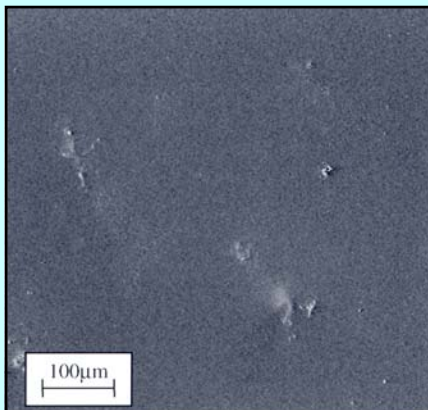
759°C / 58 h



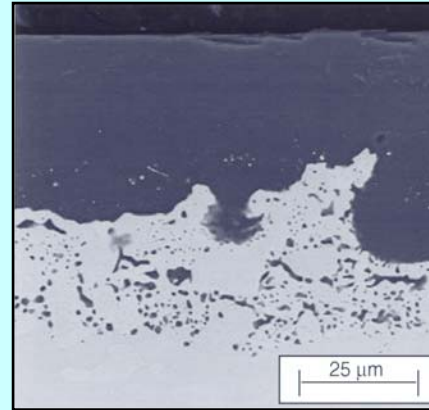
800°C / 200 h



800°C / 200 h

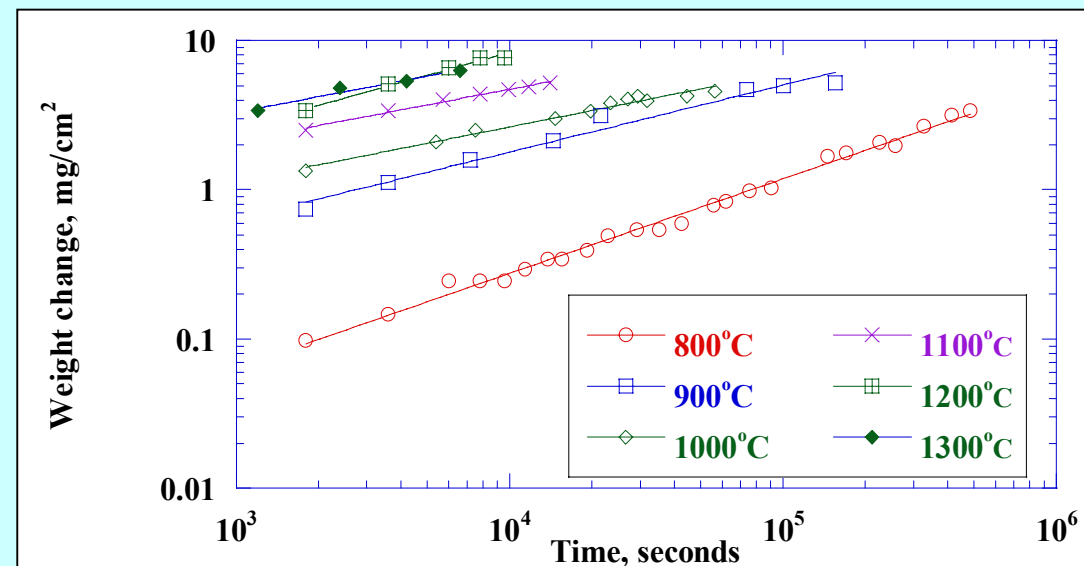
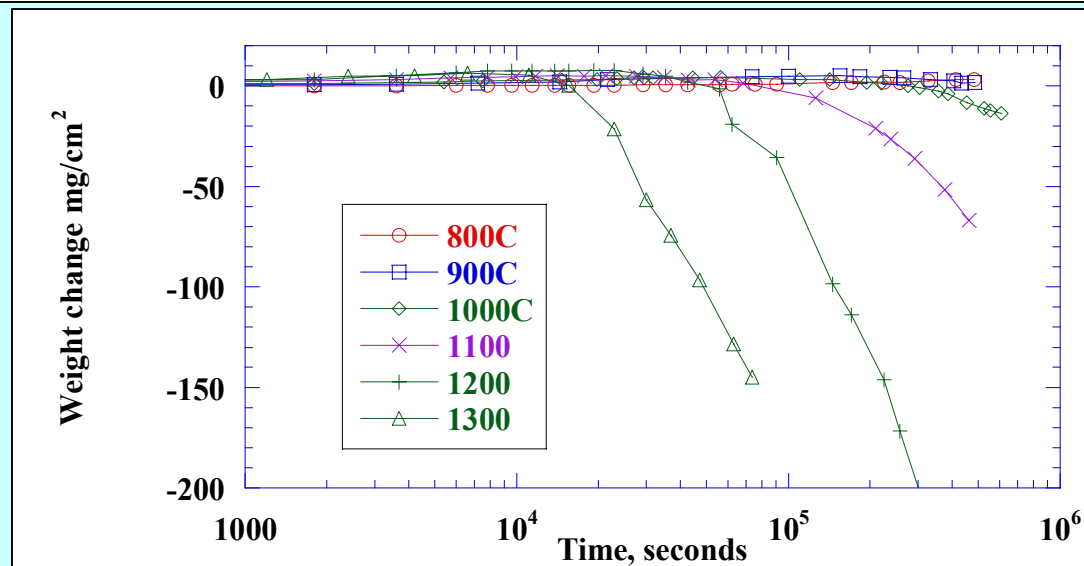


1300°C / 200 h



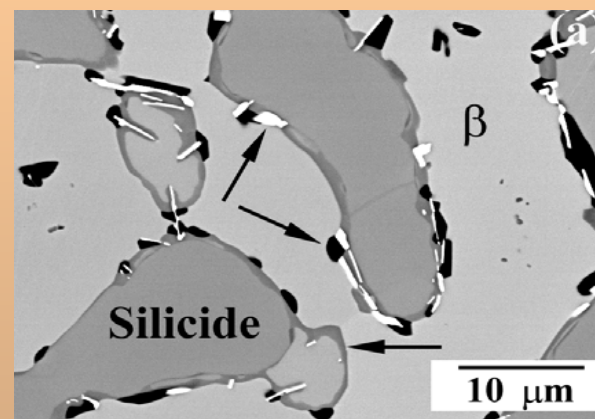
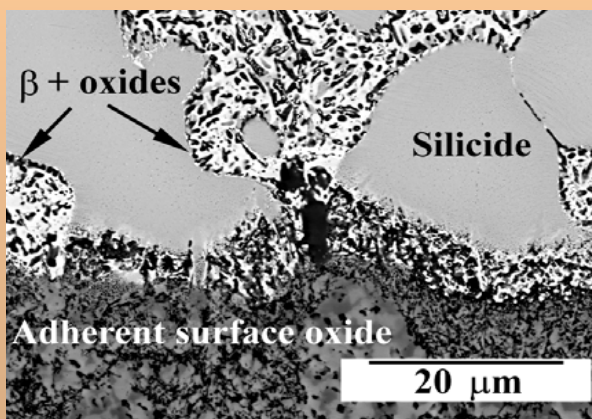
1300°C / 200 h

OXIDATION KINETICS OF Nb / Nb SILCIDE ALLOYS

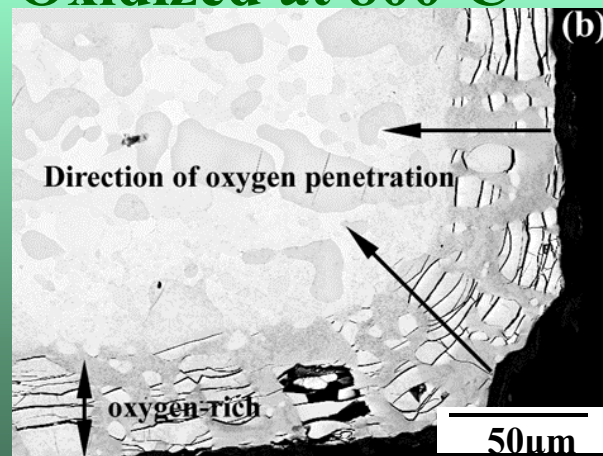
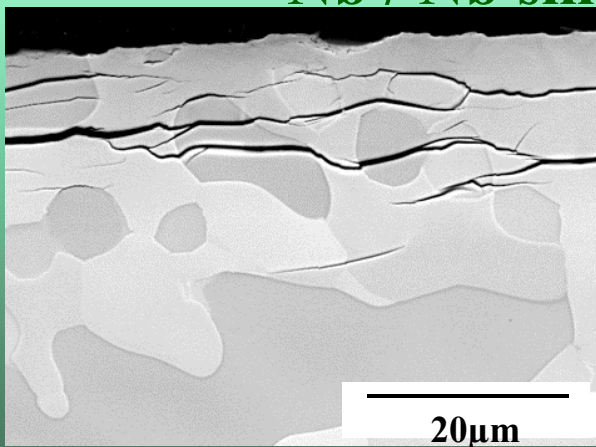


OXIDATION MICROSTRUCTURES

Nb / Nb silicide Oxidized at 1200°C



Nb / Nb silicide Oxidized at 800°C



OXIDATION MECHANISM COMPARISON

Mo-Si-B

- **Competition Between Formation of β -SiO₂ and Volatilization of Mo (MoO₃ Gas).**
 - **Below 800°C, Evaporation of Mo Dominates**
 - **Above 800°C, Protective β -SiO₂ Forms**

Nb-Ti-Cr-Si-Al-Hf-Sn

- **Formation of Complex (Nb Ti) and Si Oxides.**
- **Growth Stresses (Spallation) Limits Duration of Oxidation Protection.**
- **Low Temperature Substrate Silicide Cracking due to Internal Oxidation of Nb_{ss}.**

PROCESSING

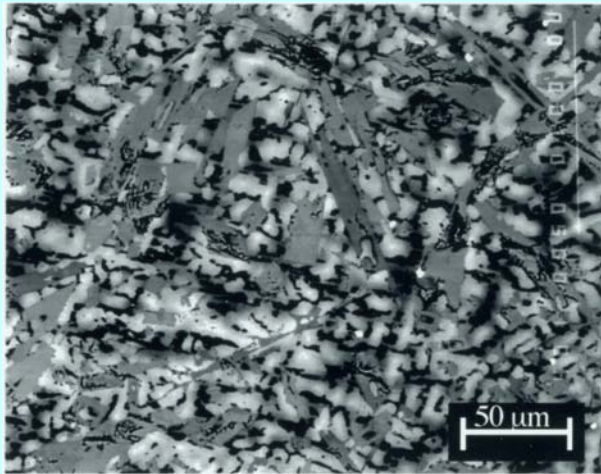
Mo-Si-B

- **Small Button Casting + Heat Treatments**
- **Prealloyed Powder → Sintering + Hipping → Hot-Extrusion**

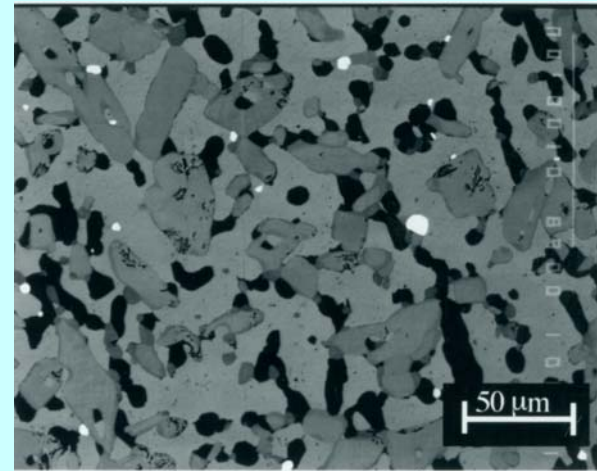
Nb-Ti-Cr-Si-Al-Hf-Sn

- **Induction Skull Melting
(6.25 cm diam. X 60 cm long ingots)
→ Hot Extrusion.**
- **Plasma Rotating Electrode Process
(PREP) Powder → Hot Extrusion.**
- **Gas-Atomization Powder
→ Hot Extrusion.**

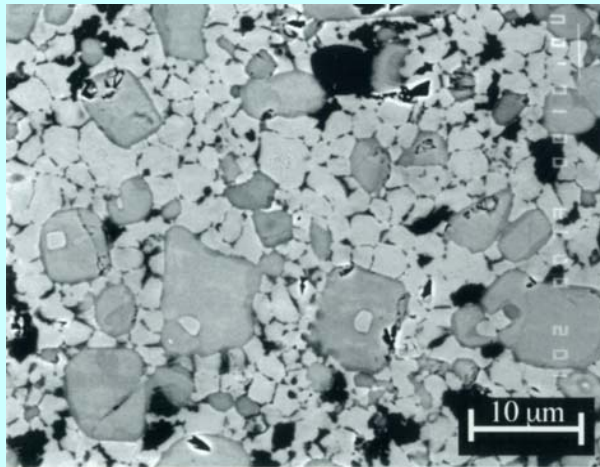
Nb – 21.4Ti – 14.3Cr – 14.4Si – 2.1Hf – 2.4Al – 1.25Sn



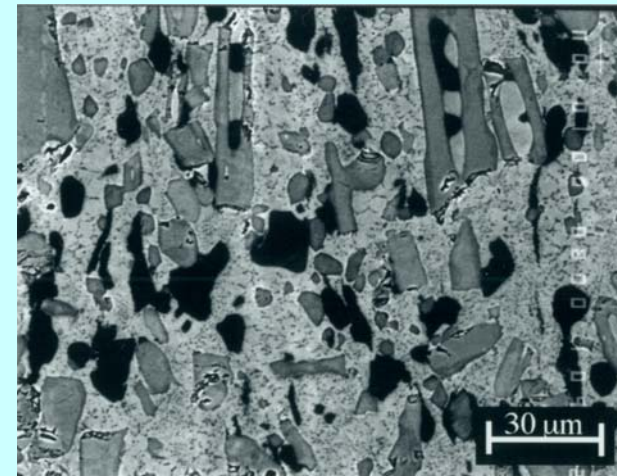
As Cast



1300⁰ C/24h + 1400⁰ C/76h



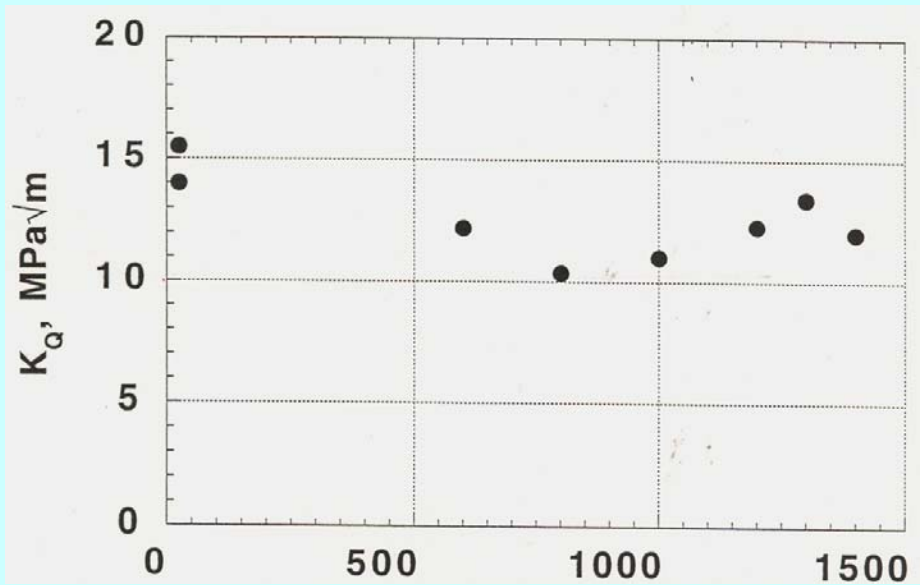
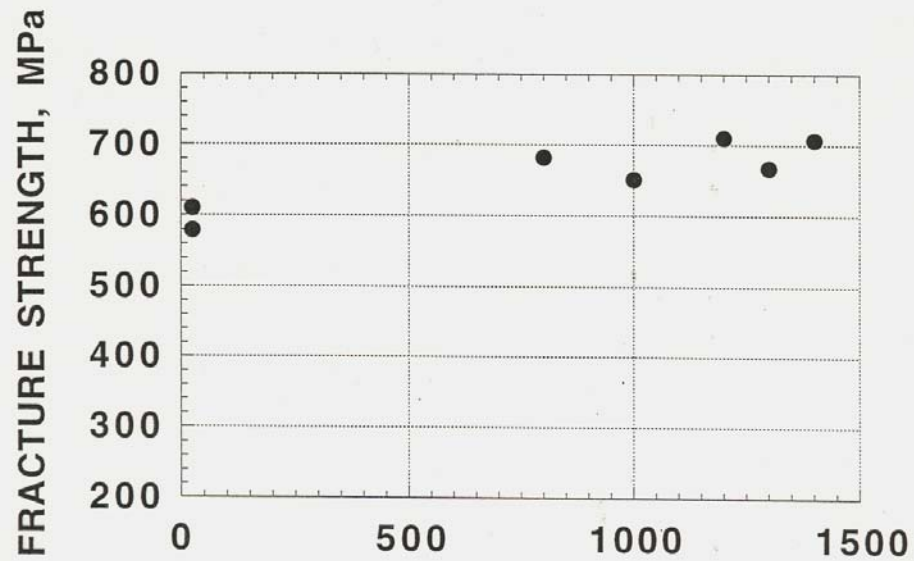
Tran.



Long.

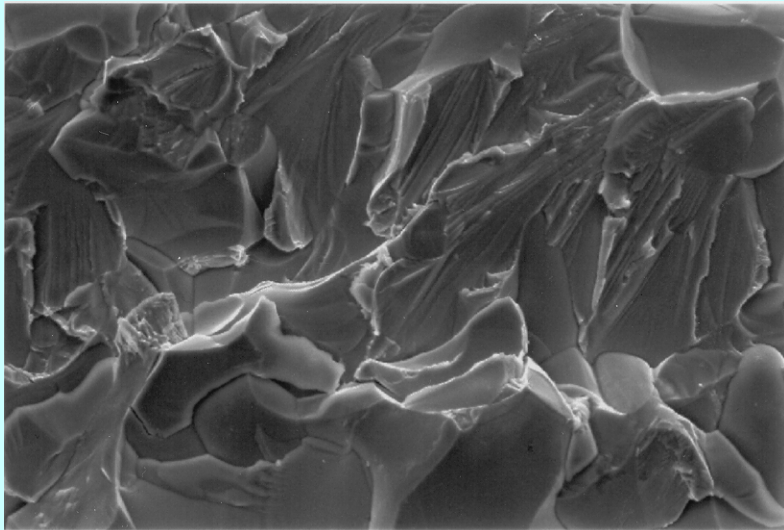
Homo. + Extruded (1350°C/6:1 R)

MECHANICAL PROPERTIES: Mo-12Si-12B

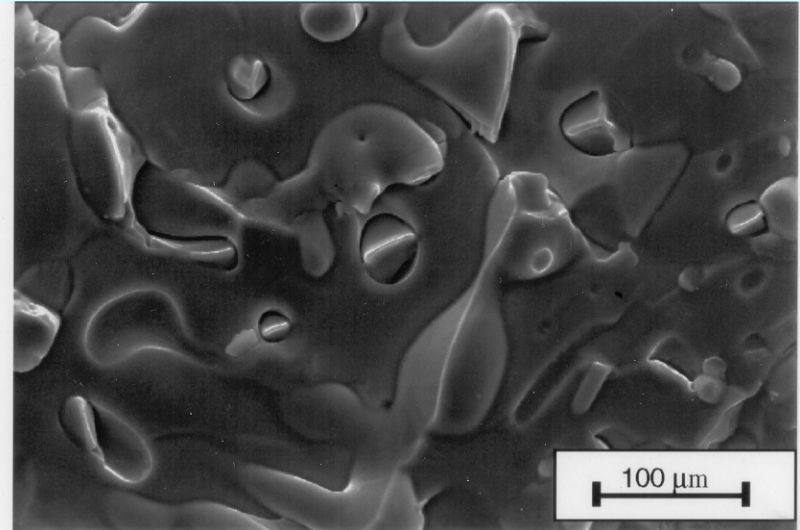


Temperature, °C

FRACTURE SURFACES: Mo – 12Si – 12B



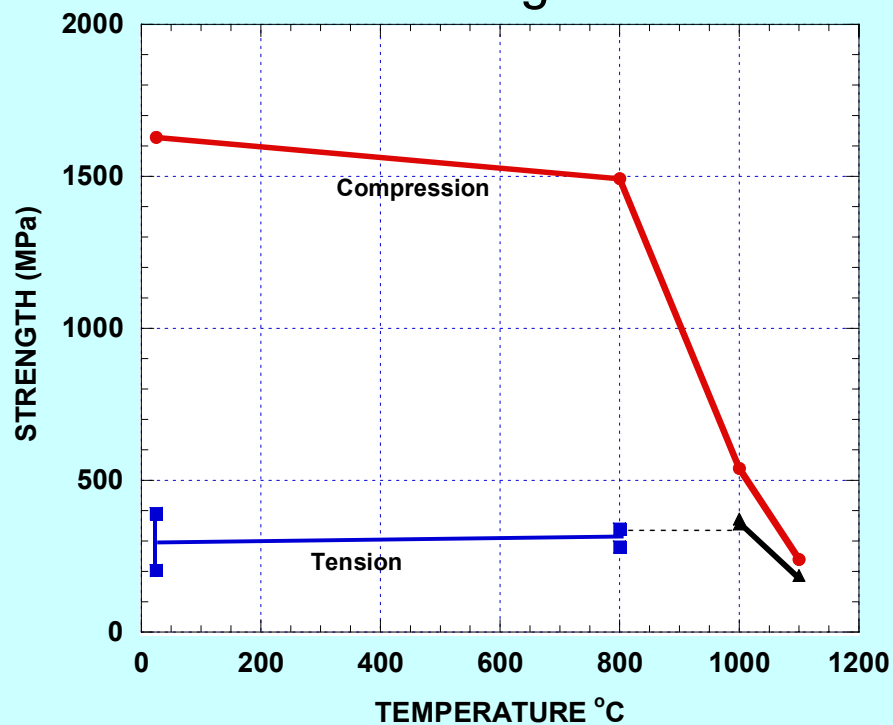
RT



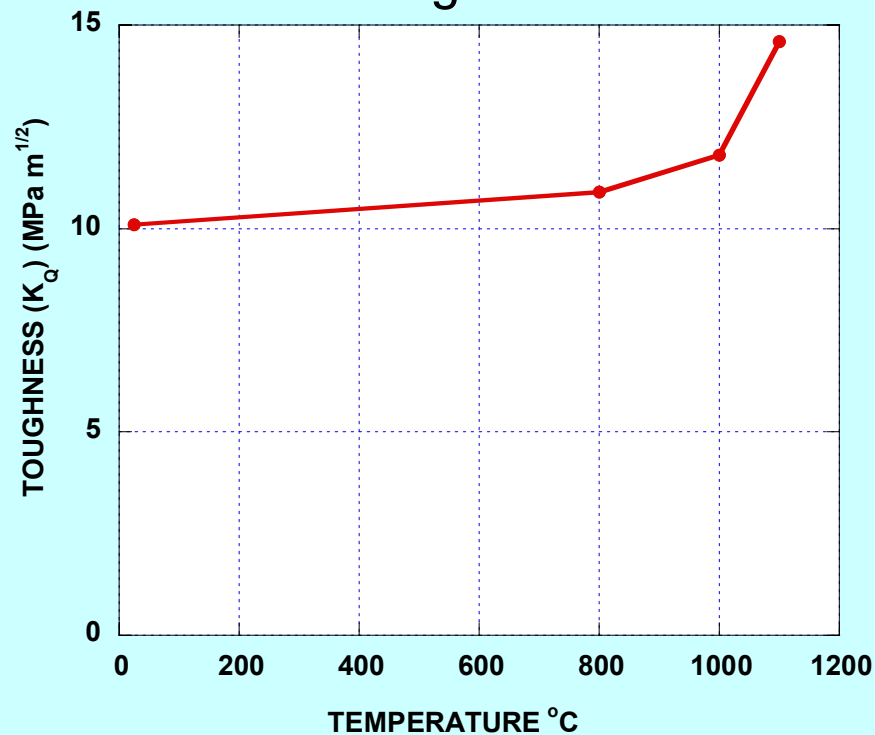
1400°C

STRENGTH AND TOUGHNESS FOR Nb / Nb SILICIDE

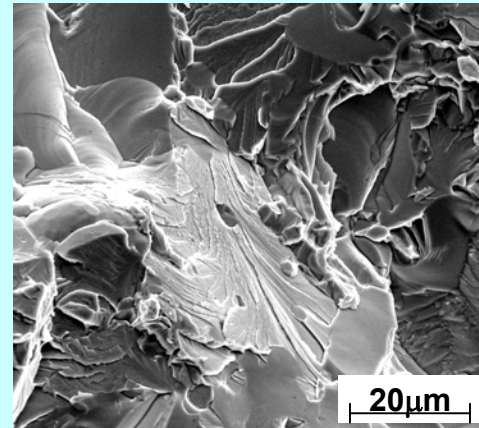
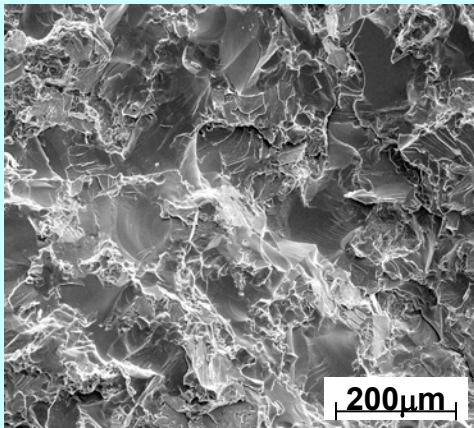
Strength



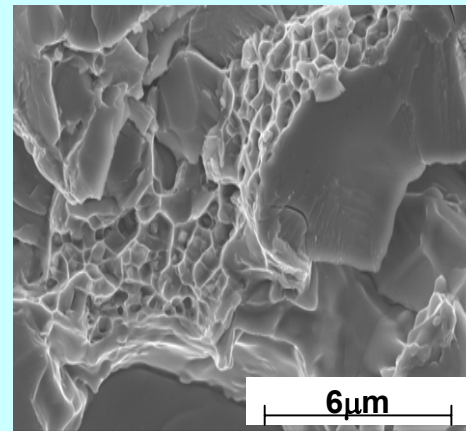
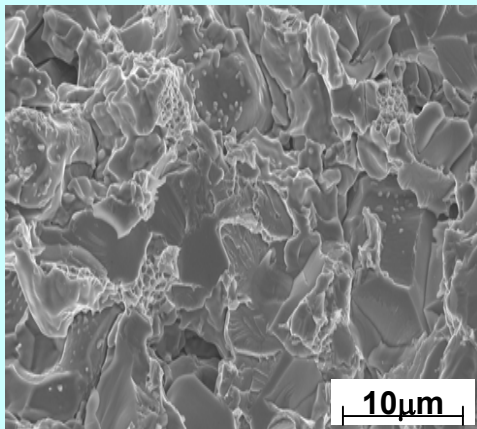
Toughness



FRACTURE MODES: Nb/Nb Silicides

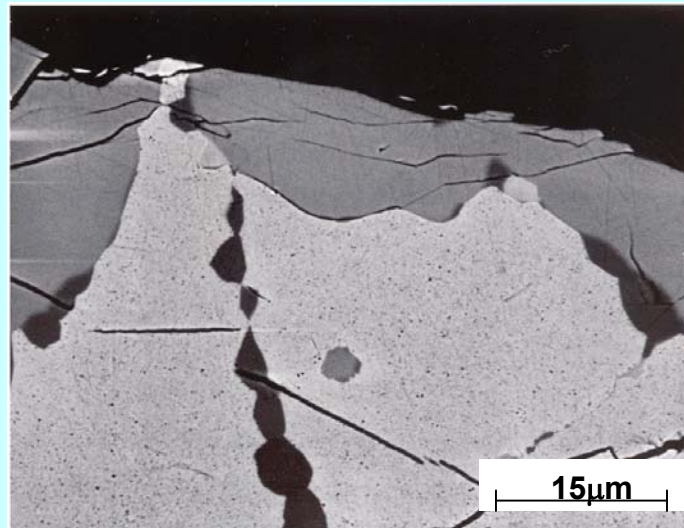
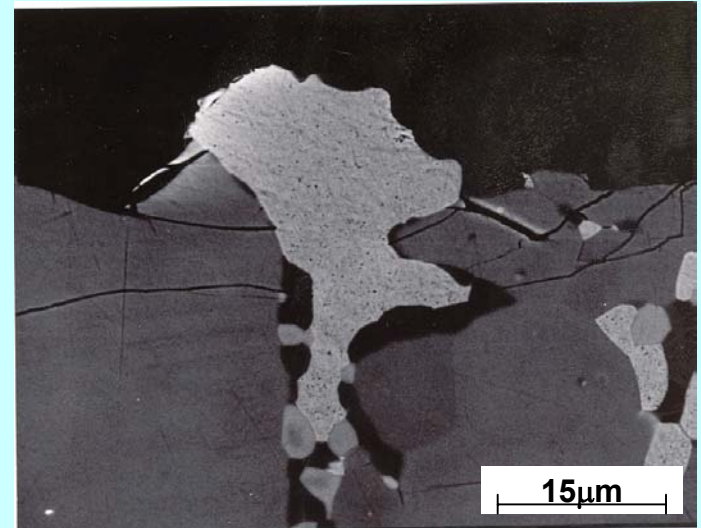
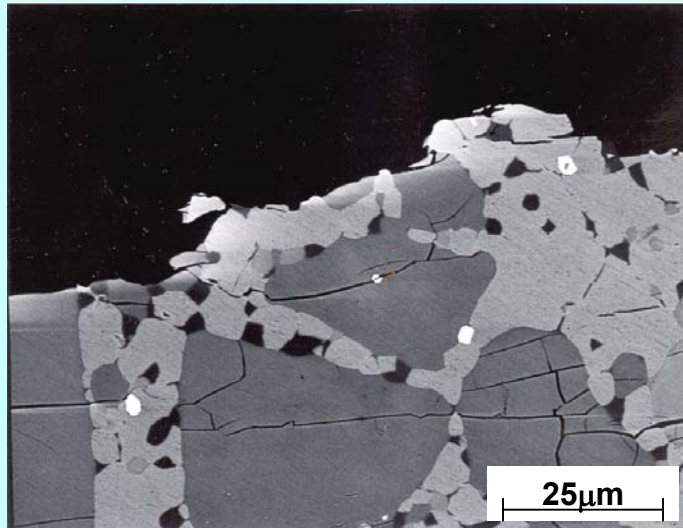


Room Temperature

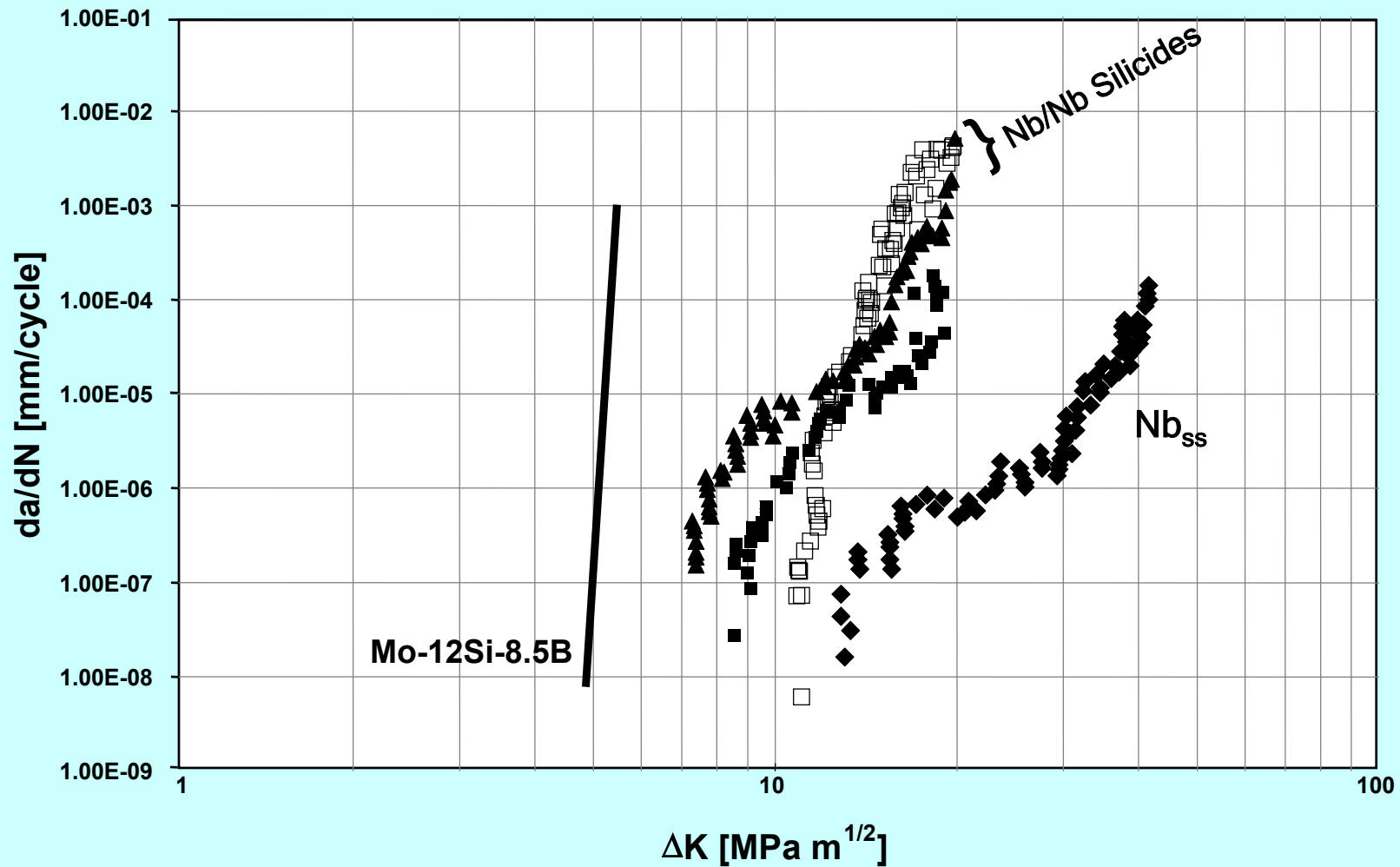


1100°C

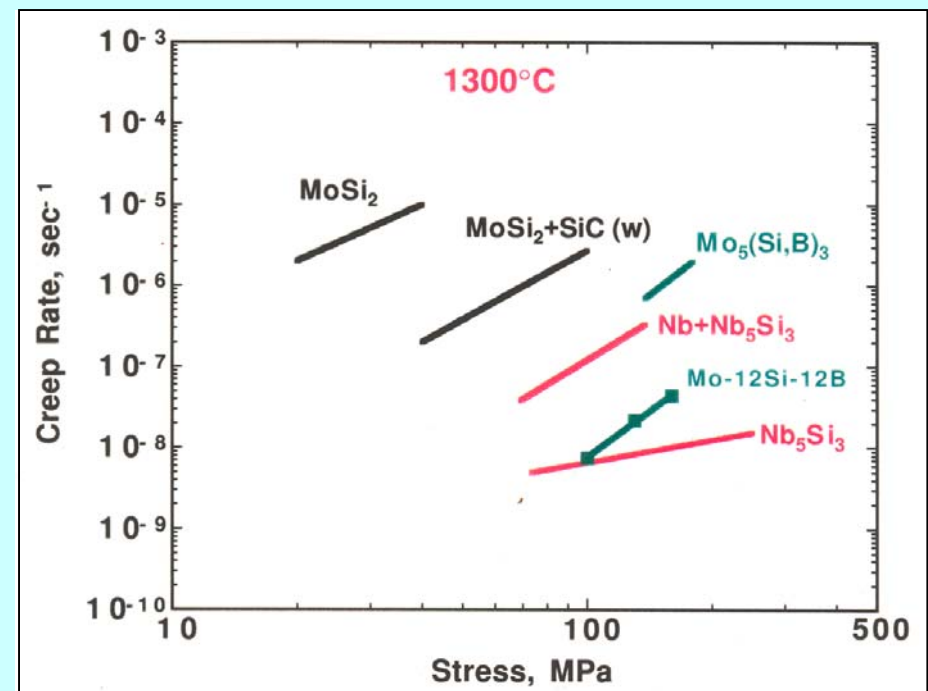
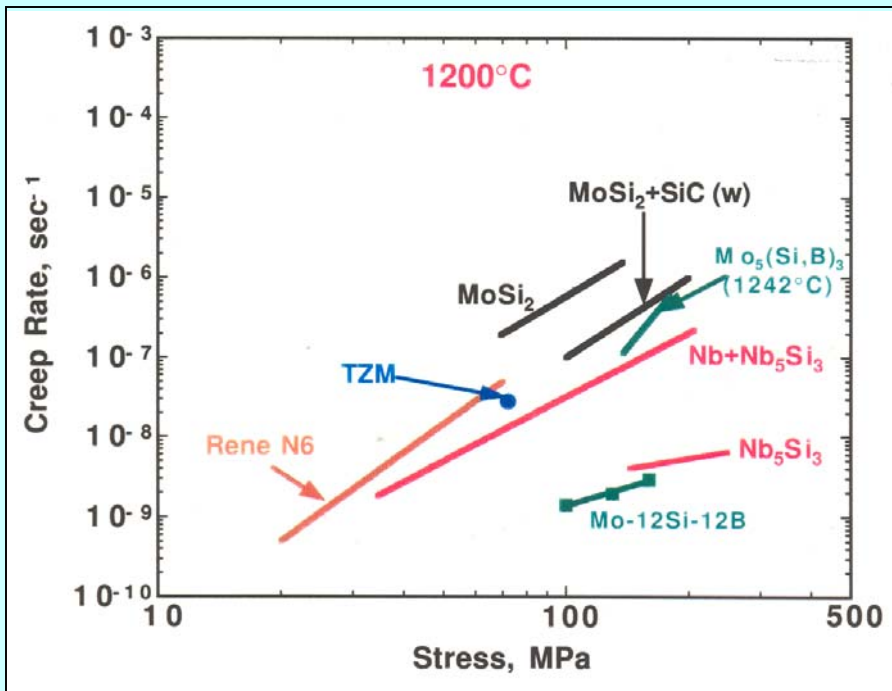
LOW TEMPERATURE DAMAGE MECHANISMS: Nb/Nb Silicides



FATIGUE CRACK GROWTH: Nb and Mo Refractory Metal / Silicides



CREEP BEHAVIOR OF Mo- AND Nb-BASED ALLOYS



SUMMARY / CONCLUSION

- Both Systems Show Promising Microstructural Stability and Reasonable Mechanical Properties i.e., Strength, Creep Resistance and Toughness.
- Both Systems are Brittle at Low Temperatures. Mo-System Exhibits Poor Fatigue and Impact Resistance. Novel Design Methods will be Needed for these Brittle/Ductile Systems
- Oxidation Resistance: Mo System Adequate from 900-1350°C. Nb-System, Limited Protection (~100 h) up to 1200°C. Both Systems will Require Coating.
- Processing and Scale-up are Major Issues for Applications.
- Comprehensive Composition / Processing / Microstructure / Property Data Still Lacking.